

**UNCLASSIFIED**

**AD NUMBER**

**AD821429**

**NEW LIMITATION CHANGE**

**TO**

**Approved for public release, distribution  
unlimited**

**FROM**

**Distribution: Further dissemination only  
as directed by Elastomers and Coatings  
Branch, [MANE], Nonmetallic Materials  
Division, Air Force Materials Laboratory,  
Wright-Patterson Air Force Base, Ohio  
45433; Aug 1967 or higher DoD authority.**

**AUTHORITY**

**AFML ltr, 7 dec 72**

**THIS PAGE IS UNCLASSIFIED**

V  
AFML-TR-66-402

AD821429

## CROSSLINKING AND FILLER REINFORCEMENT OF PERFLUOROALKYLENE TRIAZINE ELASTOMERS

T. L. GRAHAM

TECHNICAL REPORT AFML-TR-66-402

AUGUST 1967

This document may be further distributed by any holder only with specific prior approval of the Elastomers and Coatings Branch, (MANE), Nonmetallic Materials Division, Air Force Materials Laboratory, Wright-Patterson Air Force Base, Ohio 45433.

AIR FORCE MATERIALS LABORATORY  
RESEARCH AND TECHNOLOGY DIVISION  
AIR FORCE SYSTEMS COMMAND  
WRIGHT-PATTERSON AIR FORCE BASE, OHIO



DDC  
REF ID: A  
OCT 23 1967  
RIBBON SET  
A

**NOTICE**

When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related Government procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data, is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

Copies of this report should not be returned unless return is required by security considerations, contractual obligations, or notice on a specific document.

## CROSSLINKING AND FILLER REINFORCEMENT OF PERFLUOROALKYLENE TRIAZINE ELASTOMERS

*T. L. GRAHAM*

This document may be further distributed by any holder only with specific prior approval of the Elastomers and Coatings Branch, (MANE), Nonmetallic Materials Division, Air Force Materials Laboratory, Wright-Patterson Air Force Base, Ohio 45433.

FOREWORD

This report was prepared by the Elastomers and Coatings Branch, Nonmetallic Materials Division, Air Force Materials Laboratory, RTD, with Mr. T. L. Graham as project engineer. The work was initiated under Project 7340, "Nonmetallic and Composite Materials," Task 734005, "Elastomeric and Compliant Materials."

This report covers work accomplished during the period of April 1964 to September 1966.

The manuscript was released by the author September 1966 for publication as an RTD Technical Report.

This technical report has been reviewed and is approved.



J.M. KELBLE, Chief  
Elastomers and Coatings Branch  
Nonmetallic Materials Division  
AF Materials Laboratory

ABSTRACT

This exploratory compounding development program was conducted for the purpose of obtaining suitable modifiers for the crosslinking and reinforcing of triazine elastomers. The reinforcing effects of a variety of finely divided materials are compared. Data are presented showing the effect of concentration of the asbestos type filler (which yielded the most promising results) on the physical properties and thermal stabilities of a poly [perfluoropropylene (perfluoropropyl) triazine] gum and a blend comprised of equal parts of the gum and a poly [perfluoropropylene (perfluoropropyl) triazine] resin. Several organometallic and organic compounds were found to be effective crosslinking agents for poly [perfluoroctamethylene (perfluoropropyl) triazine] and n-perfluoropropylene cyanide modified poly [perfluoroctamethylene (perfluoropropyl) triazine] elastomers. The physical properties and thermal resistance of chemically vulcanized gums and asbestos reinforced vulcanizates based on the n-perfluoropropylene cyanide modified and unmodified poly [perfluoroctamethylene (perfluoropropyl) triazine] gums are discussed in detail.

This abstract may be further distributed by any holder only with specific prior approval of the Elastomers and Coatings Branch, (MANE), Nonmetallic Materials Division, Air Force Materials Laboratory, Wright-Patterson Air Force Base, 45433.

## TABLE OF CONTENTS

SECTION	PAGE
I    INTRODUCTION	1
II   EXPLORATORY FILLER REINFORCEMENT INVESTIGATIONS	2
a. Candidate Filler Reinforcing Agents Screened	2
b. Effects of Asbestine 3X Concentration on Poly [ Perfluoropropylene (perfluoropropyl) Triazine ] Gum	4
c. Effect of Asbestine 3X Concentration on Poly [ Perfluoropropylene (perfluoropropyl) Triazine ] Resin-Elastomer Blend	4
III   EXPLORATORY CHEMICAL CROSSLINKING INVESTIGATIONS	7
a. Candidate Metallic Compounds	7
b. Candidate Non-metallic Compounds and Mixtures Comprised of Metallic and Non-metallic Compounds	12
IV   CURING CHARACTERISTICS OF ADDITIONAL EXPERIMENTAL LOTS OF POLY [ PERFLUOROOCTAMETHYLENE (PERFLUOROPROPYL) TRIAZINE ] GUM	17
V   CURING CHARACTERISTICS OF EXPERIMENTAL TRIAZINE GUMS CONTAINING n-PERFLUOROPROPYLENE CYANIDE PENDANT GROUPS	18
VI   ASBESTOS REINFORCED CHEMICALLY CROSSLINKED TRIAZINE VULCANIZATES	21
VII   SUMMARY AND CONCLUSIONS	26
VIII   REFERENCES	28

## LIST OF ILLUSTRATIONS

FIGURE	PAGE
1. Thermal Stability of Triazine Gum 1927-90-6 Cured with Tetraphenyl Tin	9
2. Relative Thermal Stability of Triazine Gum 1927-90-6 Cured with Tetraphenyl Tin and Tetraphenyl Lead	11
3. Relative Thermal Stability of Triazine Gum 1927-90-6 Cured with Perfluoroglutaramidine and a Mixture of Perfluoroglutaramidine and Tetraphenyl Tin	15
4. Relative Thermal Stability of Several Chemically Cured Vulcanizates Based on n-Perfluoropropylene Cyanide Modified Triazine Gum 2227-49-1	20
5. Effect of Asbestine 3X on the Thermal Stability of Vulcanizates Based on Poly [Perfluorooctamethylene (Perfluoropropyl) Triazine] Gums	24
6. Effect of Asbestine 3X on the Thermal Stability of Vulcanizates Based on Poly [Perfluorooctamethylene (Perfluoropropyl) Triazine] Gums Containing n-Perfluoropropylene Cyanide Pendant Groups	25

## LIST OF TABLES

TABLE	PAGE
I Effect of Candidate Reinforcing Agents on the Physical Properties of Triazine Gum 1705-76	3
II Effect of Candidate Reinforcing Agents on the Physical Properties of Triazine Gum 1705-87	5
III Effect of Asbestine 3X Concentration on the Physical Properties of Triazine Gum 1705-87	5
IV Effect of Asbestine 3X Concentration on the Physical Properties of a Blend Comprised of Equal Parts of Triazine Resin 1705-69 and Triazine Gum 1705-87	6
V Tensile Properties of Triazine Gum 1927-90-6 Cured with Tetraphenyl Tin	8
VI Relative Tensile Properties of Triazine Gum 1927-90-6 Cured with Tetraphenyl Tin and Tetraphenyl Lead	10
VII Triazine Gum 1927-90-6 Compounds Containing Other Organometallic Candidate Curing Agents	10
VIII Triazine Gum 1927-90-6 Compounds Containing Non-metallic and Mixtures of Metallic and Non-metallic Compounds	13
IX Tensile Properties of Triazine Gum 1927-90-6 Cured with Perfluoroglutaramidine and a Mixture of Perfluoroglutaramidine and Tetraphenyl Tin	14
X Additional Triazine Gum 1927-90-6 Compounds Cured with Perfluoroglutaramidine	16
XI Additional Experimental Poly [Perfluoroctamethylene (perfluoropropyl) Triazine ] Gum-Curing Agent Compounds Evaluated	17
XII n-Perfluoropropylene Cyanide Modified Poly [Perfluoroctamethylene (perfluoropropyl) Triazine ] Gum-Curing Agent Compounds Evaluated	19
XIII Tensile Properties of Asbestine 3X Extended Poly [Perfluoroctamethylene (perfluoropropyl) Triazine ] Vulcanizates	22
XIV Tensile Properties of Asbestine 3X Extended Vulcanizates Based on Poly [Perfluoroctamethylene (perfluoropropyl) Triazine ] Gums Containing n-Perfluoropropylene Cyanide Pendant Groups	23

## SECTION I

### INTRODUCTION

Advanced Air Force weapon systems require elastomeric components such as O-ring seals, gasketing materials, filleting and faying surface sealants and wire insulation that can endure long periods at temperatures in excess of 400°F. Although, some commercial elastomers can withstand 400°F for an extended period, at higher temperatures the best of the available elastomers fail within a relatively short period because of embrittlement or reversion.

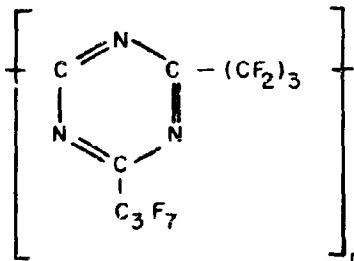
Polyperfluoroalkylene triazine is currently the prime experimental candidate elastomeric polymer under consideration for high temperature uses. Prior research has shown this type of polymer structure to have extraordinary thermal resistance at temperatures well in excess of 400°F. Samples of this polymer, after having been exposed at temperatures in the 600° to 800°F range, have retained their elastomeric characteristics. A major obstacle which has prevented this polymer from fulfilling some of the Air Force needs in the area of high temperature is the lack of a suitable means of obtaining crosslinked-reinforced products. In view of these facts internal research was initiated for the purpose of attaining the required modifiers for preparing crosslinked reinforced triazine products possessing good strength and thermal resistance at temperatures at least 100° above 400°F.

The triazine polymers used in this exploratory compounding research program were synthesized under Air Force Contracts AF33(616)-7963, AF33(657)-11252 and AF33(615)-1636 with the Hooker Chemical Corporation. Details concerning the synthesis of the polymer samples utilized in this research program are given in the reports referenced in the text of this report.

## SECTION II

### EXPLORATORY FILLER REINFORCEMENT INVESTIGATIONS

The initial polymeric triazine products, poly[perfluoropropylene (perfluoropropyl) triazine] having the idealized chemical structure shown below, were not particularly suitable for investigating crosslinking agents. The samples supplied were either hard-gelled resinous materials or stiff, partially gelled gums. Certain samples of the gum, however, could be processed and consequently were utilized for determining the relative effects of candidate reinforcing fillers.



#### POLY [ PERFLUOROPROPYLENE (PERFLUOROPROPYL) TRIAZINE ]

##### a. Candidate Filler Reinforcing Agents Screened

Several microfibrous products, a couple of carbon blacks, several metal oxide powders, a perfluorinated poly-aromatic resinous product, and a few mineral fillers were screened in seeking suitable materials for the reinforcement of the triazine polymer. Because of the limited supply of a given sample of polymer, two separate samples of the poly[perfluoropropylene (perfluoropropyl) triazine] gum (Lots 1705-76\* and 1705-87) (Reference 1) were involved in screening the selected candidate reinforcing agents. Since the presence of water was suspected of being undesirable, the fillers were heat treated to remove absorbed moisture. Compounds containing 15 phr of the selected fillers (in the case of Cab-O-Sil, 10 phr) were prepared for the investigation. All of the candidate reinforcing agents were incorporated and dispersed in the elastomer using a micro-mill.

The slabs required for determining tensile properties were formed by two different techniques. Slabs for the compounds in the first seven columns in Table I were obtained by banding the stock onto the micro-mill and cutting it off after forming a smooth band. This technique was used initially, since samples compression molded at 300°F, shrank and cracked upon cooling in the opened mold. It was later discovered, however, that this problem could be circumvented by compression molding at room temperature. Consequently, all subsequent samples involved in the exploratory filler screening investigations were compression molded at room temperature.

Since research (Reference 2) preceding these investigations had demonstrated that some degree of cure could be obtained by simply heating the polymer in air at an elevated temperature, these compounds were heat treated in air for 16 hours at 600°F.

\*Lot 1705-76 is not referenced; Synthesis procedures for the preparation of Lot 1705-76 same as Lot 1705-67.

**TABLE I**  
**EFFECT OF CANDIDATE REINFORCING AGENTS ON THE PHYSICAL  
 PROPERTIES OF TRIAZINE GUM 1705-76**

	100	100	100	100(1)	100	100	100	100	100	100	100	100	100	100
Triazine 1705-76	-	15	-	-	-	-	-	-	-	-	-	-	-	-
Carbon Wool 3B1	-	-	15	-	-	-	-	-	-	-	-	-	-	-
Wilcerbo	-	-	-	15	-	-	-	-	-	-	-	-	-	-
EPC Black	-	-	-	-	15	-	-	-	-	-	-	-	-	-
Alen C	-	-	-	-	-	15	-	-	-	-	-	-	-	-
Pyrax A	-	-	-	-	-	-	15	-	-	-	-	-	-	-
Mineral Black #35	-	-	-	-	-	-	-	15	-	-	-	-	-	-
Cat-C-Sil	-	-	-	-	-	-	-	-	15	-	-	-	-	-
Asbestos Superfine	-	-	-	-	-	-	-	-	-	10	-	-	-	-
Asbestine 3X	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Paxar V-2	-	-	-	-	-	-	-	-	-	-	15	-	-	-
Asbestine Fine Texture	-	-	-	-	-	-	-	-	-	-	-	15	-	-
Superglas 150C	-	-	-	-	-	-	-	-	-	-	-	-	15	-
<u>Heat Treated 16 Hours @ 600° F</u>														
Tensile Strength, psi	90	190	195	410	-	235	260	90	165	180	170	170	175	130
Elongation, %	80	65	80	70	-	85	90	115	100	80	95	40	85	85
Hardness, Shore A, Pl.	34	50	52	74	-	46	47	37	60	55	50	70	50	55
Weight Loss, % (2)	-	-	-	-	-	-	-	18.8	20.6	19.6	18.9	19.3	18.8	19.9
<u>Aged in Air 72 Hours @ 700° F</u>														
Tensile Strength, psi	40	235	275	485	-	305	260	100	-(3)	425	295	275	375	250
Elongation %	100	45	75	20	-	75	95	110	-	75	85	25	90	70
Hardness, Shore A, Pl.	72	82	93	91	-	96	70	39	-	62	55	80	60	69
Weight Loss, % (2)	-	-	-	-	-	-	-	22.2	39.9	18.2	14.8	18.2	15.1	35.3

(1) The calendered sample was spotted with cracks.

(2) Calculation based on weight of polymer.

(3) Extremely brittle.

Tensile data obtained on the thermally cured products before and after aging 72 hours at 700°F are summarized in Tables I and II. As these results show, all of the finely divided materials to varying degrees augmented the strength of these elastomeric triazine gums. The results, however, indicated the microfibrous asbestos type extender to be the best choice. With these materials, marginal improvements in the thermal stability and significant increases in the tensile strength were obtained at nominal reductions in elongation. Although some of the other candidate extenders exhibited an equal or slightly greater reinforcing effect than the asbestos products, elongation and/or thermal stability were sacrificed.

b. Effects of Asbestine 3X Concentration on Poly[Perfluoropropylene (perfluoropropyl) Triazine]Gum

Additional samples of the poly[perfluoropropylene (perfluoropropyl) triazine]gum (Lot 1705-87) were extended with low to moderately high loadings (10 to 40 parts) of Asbestine 3X to further investigate the merits of the microfibrous asbestos type material.

The data obtained on samples heat treated for 16 hours at 600°F and those obtained after 72 hours aging at 700°F are presented in Table III. Increases in tensile strength were realized with an increase in the concentration of this asbestos microfibrous material. For the sake of preserving thermal stability and elongation, however, these results indicate that the concentration of asbestos should be limited to about 20 phr.

c. Effect of Asbestine 3X Concentration on Poly[Perfluoropropylene (perfluoropropyl) Triazine] Resin-Elastomer Blend

The Hooker Chemical Corporation obtained an elastomeric composite having a tensile strength of 1000 psi by blending a poly[perfluoropropylene (perfluoropropyl) triazine]resinous product with a poly[perfluoropropylene (perfluoropropyl) triazine]gum. As an extension of the Hooker work, research was conducted to investigate the merits of using asbestos filler in conjunction with a triazine resin for reinforcing triazine elastomers.

For this investigation compounds containing 0, 15, 30 and 45 phr of Asbestine 3X were prepared using a polymer blend comprised of equal parts of poly[perfluoropropylene (perfluoropropyl) triazine]gum (Lot 1705-87) and poly[perfluoropropylene (perfluoropropyl) triazine]resin (Lot 1705-69) (Reference 1).

A stability problem was encountered with the original compounds. When heated to cure at 600°F these samples sponged. Investigation of sponging revealed that it had been caused by volatile (easily sublimed) material released by the resinous triazine product. However, satisfactory thermally cured compounds were obtained using the fraction of this resinous polymer that was not volatile at 100°C under vacuum (2 to 3mm Hg).

The resin in conjunction with the asbestos filler yielded products of substantial strength (565 to 900 psi). Elongation, however, was sacrificed and the compounds proved to be extremely unstable (see Table IV).

TABLE II

## EFFECT OF CANDIDATE REINFORCING AGENTS ON THE PHYSICAL PROPERTIES OF TRIAZINE GUM 1705-87

Triazine 1705-87	100	100	100	100	100	100
Perfluoropolyphenylene #2112	-	15	-	-	-	-
Amorphous Graphite #7	-	-	15	-	-	-
Crystalline Graphite #7	-	-	-	15	-	-
Tipersu <sup>1</sup>	-	-	-	-	15	-
Baymal	-	-	-	-	-	15
<u>Heat Treated 16 Hours @ 600°F</u>						
Tensile Strength, psi	90	195	340	345	300	180
Elongation, %	130	95	70	80	50	70
Hardness, Shore A, Pts.	40	60	63	68	70	66
Weight Loss, % (1)	16.4	19.3	21.8	19.0	25.4	26.5
<u>Aged in Air 72 Hours @ 700°F</u>						
Tensile Strength, psi	110	95	425	550	- (2)	265
Elongation, %	120	130	50	45	-	30
Hardness, Shore A, Pts.	50	50	94	90	-	81
Weight Loss, % (1)	36.0	52.3	73.3	41.1	94.6	18.3

(1) Calculation based on weight of polymer.

(2) Extremely brittle.

TABLE III

## EFFECT OF ASBESTINE 3X CONCENTRATION ON THE PHYSICAL PROPERTIES OF TRIAZINE GUM 1705-87

Triazine 1705-87	100	100	100	100	100	100
Asbestine 3X	-	10	15	20	30	40
<u>Heat Treated 16 Hours @ 600°F</u>						
Tensile Strength, psi	90	150	185	170	230	285
Elongation, %	130	125	130	105	95	70
Hardness, Shore A, Pts.	40	55	55	55	70	75
Weight Loss, % (1)	16.4	18.6	17.9	18.3	17.0	16.2
<u>Aged in Air 72 Hours @ 700°F</u>						
Tensile Strength, psi	110	185	345	310	415	390
Elongation, %	120	80	100	95	65	30
Hardness, Shore A, Pts.	50	65	70	70	85	90
Weight Loss, % (1)	36.0	41.3	31.7	34.2	43.9	47.7

(1) Calculation based on weight of polymer.

TABLE IV

EFFECT OF ASBESTINE 3X CONCENTRATION ON THE PHYSICAL  
 PROPERTIES OF A BLEND COMPRISED OF EQUAL PARTS OF  
 TRIAZINE RESIN 1705-69 AND TRIAZINE GUM 1705-87

Triazine Blend (1)	100	100	100	100
Asbestine 3X	-	15	30	45
<u>Heat Treated 16 Hours @ 600°F</u>				
Tensile Strength, psi	565	710	810	900
Elongation, %	65	50	30	30
Hardness, Shore A, Pts.	80	91	97	97
Weight Loss, % (2)	17.2	19.8	21.2	22.1
<u>Aged in Air 72 Hours @ 700°F</u>				
Tensile Strength, psi	360	165	- (3)	- (3)
Elongation, %	90	15	-	-
Hardness, Shore A, Pts.	80	88	-	-
Weight Loss, (2)	40.3	68.6	75.2	-

(1) 50/50: Triazine gel 1705-87/Triazine gel 1705/67.

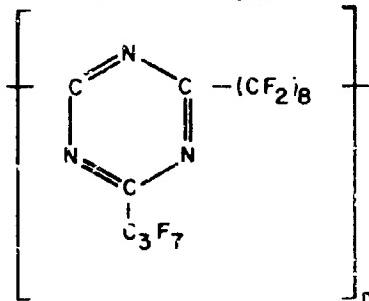
(2) Calculation based on weight of polymer.

(3) Extremely brittle.

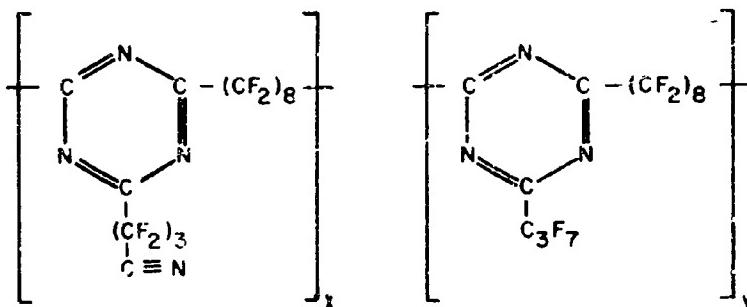
### SECTION III

#### EXPLORATORY CHEMICAL CROSSLINKING INVESTIGATIONS

Samples of poly [perfluorooctamethylene (perfluoropropyl) triazine] and n-perfluoropropylene cyanide modified poly [perfluorooctamethylene (perfluoropropyl) triazine] gums (their idealized structures are shown below) were supplied at various intervals during this program and were used for crosslinking investigations. These later samples, although also partially gelled were more elastic and were more easily processed than the previous samples of gelled poly [perfluoropropylene (perfluoropropyl) triazine] gum.



**POLY [PERFLUOROOCTAMETHYLENE (PERFLUOROPROPYL) TRIAZINE]**



**N-PERFLUOROPROPYLENE CYANIDE MODIFIED POLY [PERFLUOROOCTAMETHYLENE (PERFLUOROPROPYL TRIAZINE)]**

A number of organometallic and non-metallic compounds and several mixtures comprised of metallic and non-metallic organic compounds were screened in seeking suitable curing agents for the above types of triazine gums. The selected candidate compounds were initially screened using poly [perfluorooctamethylene (perfluoropropyl) triazine] gum (Lot 1927-90-6).

**a. Candidate Metallic Compounds**

The metallic compounds evaluated are listed below:

- Tetraphenyl Tin
- Tetraphenyl Lead
- Tetraphenyl Silicon
- Tetraphenyl Germanium
- Perfluorotetraphenyl Tin
- Hexamethyl Borazole

Triphenyl Stibine  
 E-Triphenyl Borazole  
 Dibutyl Tin Mercaptopropionate  
 Dibutyl Tin Bis(Lauryl Mercaptide)  
 Dibutyl Tin Bis(Isooctyl Thioglycolate)  
 Chromium Trifluoroacetate

Tetraphenyl Tin (which Hooker had shown in an earlier experiment to be an effective curing agent for the n-perfluoropropylene cyanide modified poly[perfluorooctamethylene (perfluoropropyl) triazine] gum was also effective for curing the above sample of poly[perfluorooctamethylene (perfluoropropyl) triazine] gum. The rate of reaction was very slow and was not observed to be significantly influenced by curing agent concentration. Increases in tensile strength were realized with increased concentration of tetraphenyl tin (Table V) but the tensile properties of the vulcanizates obtained using 2.5, 5 and 10 phr tetraphenyl tin were in general low. As shown in Figure 1 the vulcanizates proved to be less thermally stable than the uncured gum in the 580° to 630°F temperature range. In contrast to the 16 percent weight lost by the uncured gum in a 400-hour period, the tetraphenyl tin vulcanizates lost from 47 to 63 percent weight during this same period.

TABLE V  
 TENSILE PROPERTIES OF TRIAZINE GUM 1927-90-6  
 CURED WITH TETRA-PHENYL TIN

	100	100	100
Tetraphenyl Tin	2.5	5	10
<u>Initial Physical Properties</u>			
<u>Press Cured 20 Hours. @ 320°F</u>			
Tensile Strength, psi	245	285	325
Elongation, %	405	495	385
<u>Heated in Air 16 Hours @ 580° to 630°F</u>			
Tensile Strength, psi	180	125	115
Elongation, %	315	230	190
<u>Heated in Air an Additional 72 Hours @ 580° to 630°F</u>			
Tensile Strength, psi	125	110	115
Elongation, %	310	250	160

Tetraphenyl lead was the only other organometallic compound evaluated which yielded a structurally sound vulcanizate based on this particular sample of triazine gum. As the tensile data Table VI indicate, it was slightly less effective than tetraphenyl tin and yielded (see Figure 2) a relatively unstable vulcanizate.

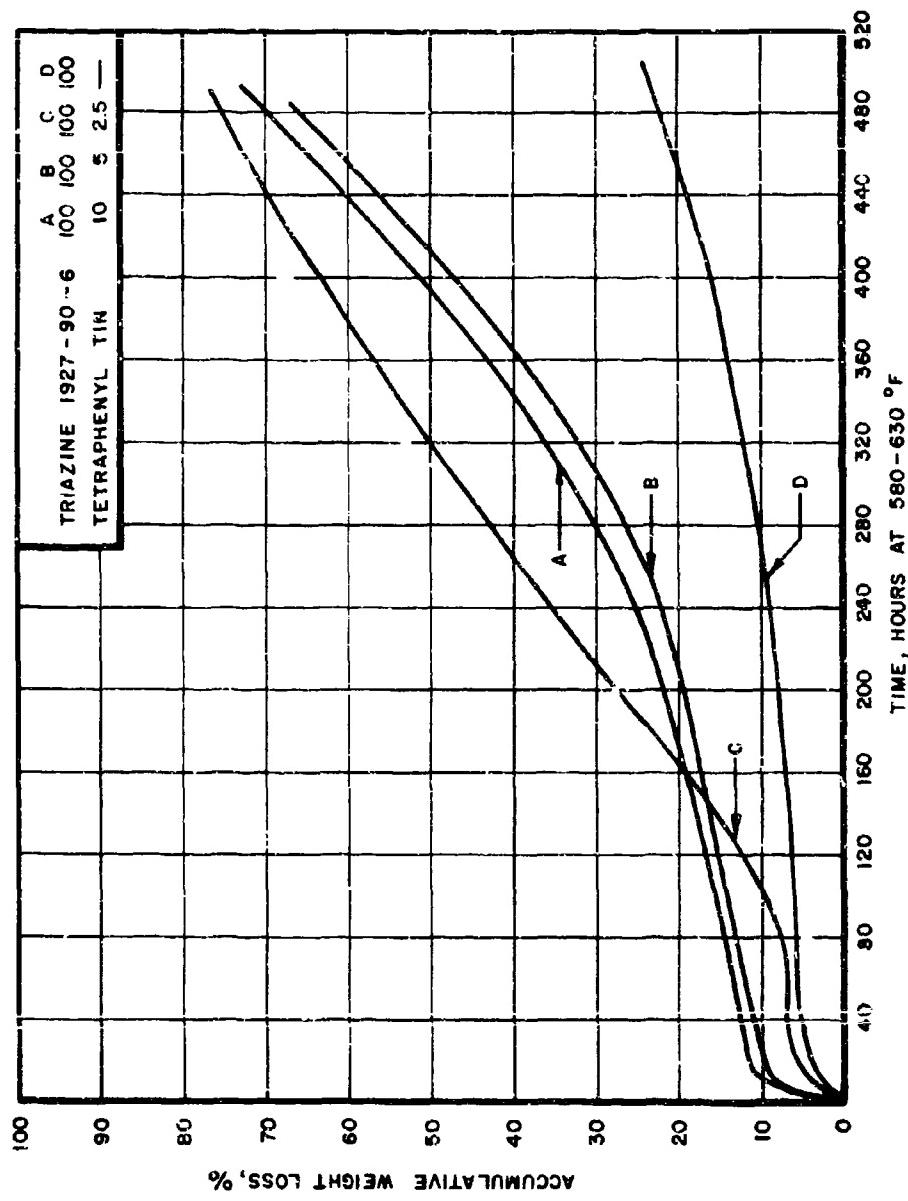


Figure 1. Thermal Stability of Triazine Gum 1927-90-6 Cured with Tetraphenyl Tin

TABLE VI

RELATIVE TENSILE PROPERTIES OF TRIAZINE GUM 1927-90-6  
CURED WITH TETRAPHENYL TIN AND TETRAPHENYL LEAD

Triazine 1927-90-6	100	100
Tetraphenyl Tin	5	-
Tetraphenyl Lead	-	5
<u>Initial Physical Properties</u>		
<u>Press Cured 20 Hours @ 320°F</u>		
Tensile Strength, psi	285	235
Elongation, %	495	295
<u>Heated in Air 16 Hours @ 580° to 630°F</u>		
Tensile Strength, psi	125	160
Elongation, %	230	390
<u>Heated in Air an Additional 72 Hours</u>		
Tensile Strength, psi	110	135
Elongation, %	250	385

Adverse effects were obtained with perfluorotetraphenyl tin and hexamethyl borazole. These candidate curing agents appeared to decompose the gum. Triphenyl stibine proved to be inert. Partially cured porous products were obtained with the remaining candidate curing agents. The compounds prepared and the curing conditions used in the unsuccessful attempts to obtain crosslinked products are given in Table VII.

TABLE VII

TRIAZINE GUM 1927-90-6 COMPOUNDS CONTAINING OTHER  
ORGANOMETALLIC CANDIDATE CURING AGENTS

	Parts/100 Parts Triazine Elastomer								
Perfluorotetraphenyl Tin	5								
Triphenyl Stibine		5							
B-Triphenyl Borazole			5						
Hexamethyl Borazole				5					
Dibutyl Tin Mercaptopropionate					2.1				
Dibutyl Tin Bis (lauryl mercaptide)						2.1	2.1		
Dibutyl Tin Bis (isooctyl thioglycolate)								2.1	
Chromium Trifluoroacetate									5
Press Cure Time @ 320°F, Hours.	20	20	20	20	18	0.5	16.5	17.5	1

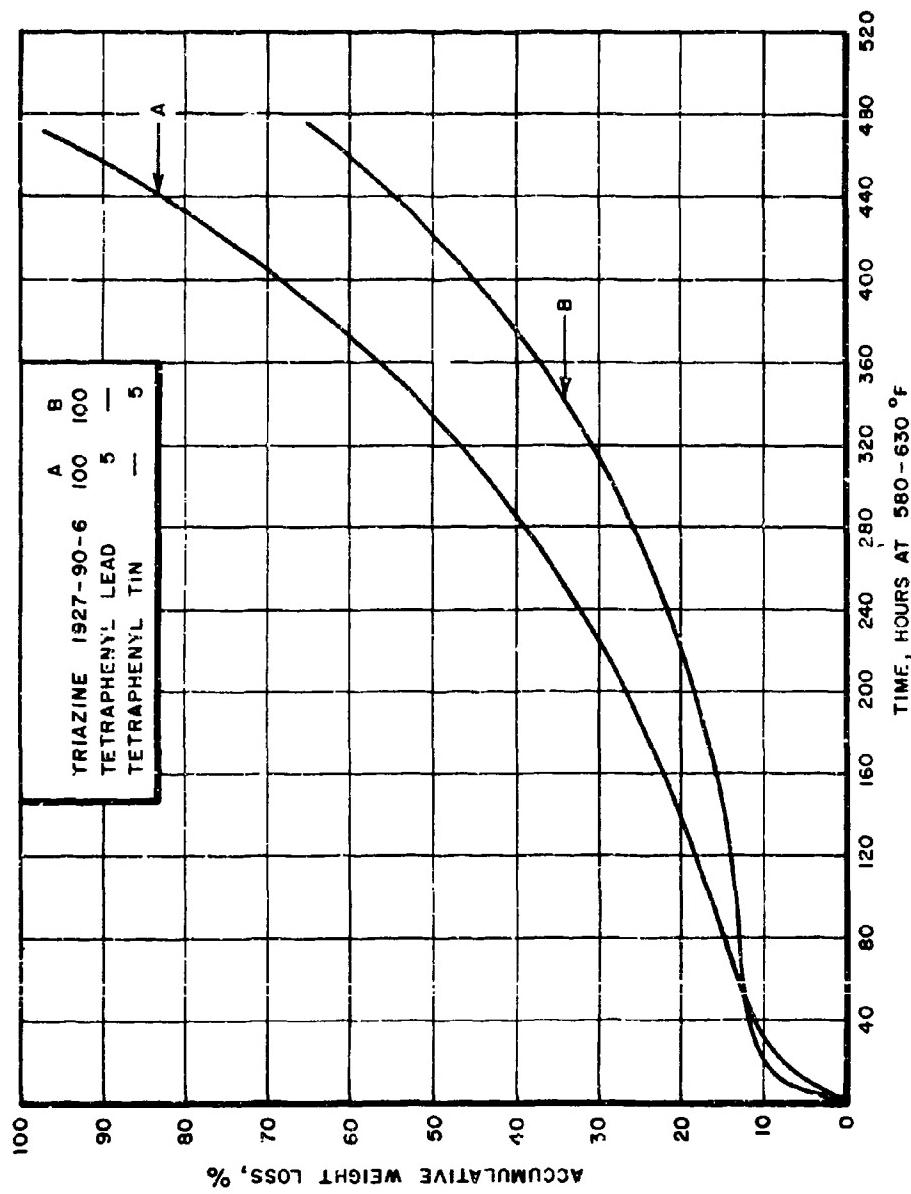


Figure 2. Relative Thermal Stability of Triazine Gum 1927-90-6 Cured with Tetraphenyl Tin and Tetraphenyl Lead

b. Candidate Non-metallic Compounds and Mixtures Comprised of Metallic and Non-metallic Compounds

The non-metallic organic compounds and compound mixtures screened are listed below:

1,3 Perfluorophenylene Diamine  
Perfluoroglutarimidine  
Perfluoroglutarimidine-Tetraphenyl Tin  
Perfluorosebaconitrile-Tetraphenyl Tin  
Perfluoroglutaronitrile-Hexabutyl Distannoxane  
Perfluorosebacamidine  
 $\alpha, \alpha$ -Diphenyl-m-Xylylidene Polymer  
Dicyclopentadiene Dioxide  
Triphenyl Phosphine

With the exception of perfluoroglutarimidine and the perfluoroglutarimidine-tetraphenyl tin mixture, the candidates listed above did not yield satisfactory vulcanizates. Perfluorophenylene diamine, triphenyl phosphine, dicyclopentadiene dioxide and  $\alpha, \alpha$ -diphenyl-m-xylylidene polymer appeared to degrade the triazine gum. Perfluoroglutaronitrile-hexabutyl distannoxane and perfluorosebaconitrile-tetraphenyl tin mixtures proved to be inactive. Undercured products spotted with voids were obtained with perfluorosebacamidine. The compositions of the compounds and the thermal treatments used in the unsuccessful attempts to obtain cured products are given in Table VII.

Rapid cures were obtained with perfluoroglutarimidine and a mixture comprised of equal parts of perfluoroglutarimidine and tetraphenyl tin. Tensile properties of these vulcanizates (Table IX) were similar to those of the tetraphenyl tin vulcanizates (shown in Table V). The perfluoroglutarimidine vulcanizate had a lower rate of weight loss than the gum cured with the mixture, (Figure 3) but became spotted with numerous tiny pinholes within the first several hours of exposure at 580° to 630°F.

In further attempting to obtain a thermally stable perfluoroglutarimidine vulcanizate a number of additional compounds were prepared wherein the concentration of curing agent as well as the cure time were varied over a broad range. A couple of vulcanizates were also prepared using purified (freshly sublimed) perfluoroglutarimidine. As an additional precaution the gum used in the preparation of one of these samples was also preheated at 600°F for approximately 472 hours to remove volatiles.

These modifications did not produce the expected improvement. Within 48 hours or less at 600°F these additional perfluoroglutarimidine vulcanizates (Table X) lost their structural integrity.

TABLE VIII  
TRAZINE GUM 1927-90-6 COMPOUNDS CONTAINING NON-METALLIC AND MIXTURES  
OF METALLIC AND NON-METALLIC COMPOUNDS AS CANDIDATE CURING AGENTS

	Parts/100 Parts Trazone Elastomer					
E	4 drops	5	several drops	several drops	1	10
1,3 Perfluorophenylene Diamine						
Perfluorosebaconitrile						
Tetraphenyl Tin						
Perfluoroglutaronitrile						
Hexabutyl Distannoxane						
Perfluorosebacamidine						
$\alpha, \alpha$ Diphenyl-m-xylylidene Polymer						
Dicyclopentadiene Dioxide						
Triphenyl Phosphine						
Press-Cure Time @ 320° F, hours	20	1	1	1 1/4	4	1 1/2
Press-Cure Time @ 600° F, hours					-	3 1/2
						64 1/2
						64 1/2

TABLE IX

TENSILE PROPERTIES OF TRIAZINE GUM 1927-90-6 CURED WITH  
 PERFLUOROGLUTARIMIDINE AND A MIXTURE OF PERFLUORO-  
 GLUTARIMIDINE AND TETRAPHENYL TIN

Triazine 1927-90-6	100	100
Perfluoroglutarimidine	5	5
Tetraphenyl Tin	5	-
<b>Initial Physical Properties</b>		
Press Cure Time @ 320° F, Min.	20	4
Tensile Strength, psi	220	230
Elongation, %	480	590
<b>Heated in Air 16 Hours @ (580° To 630° F)</b>		
Tensile Strength, psi	265	- (1)
Elongation, %	320	-
<b>Heated in Air an Additional 72 Hours @ (580° To 630° F)</b>		
Tensile Strength, psi	135	-
Elongation, %	245	-

(1) Weight Loss data only

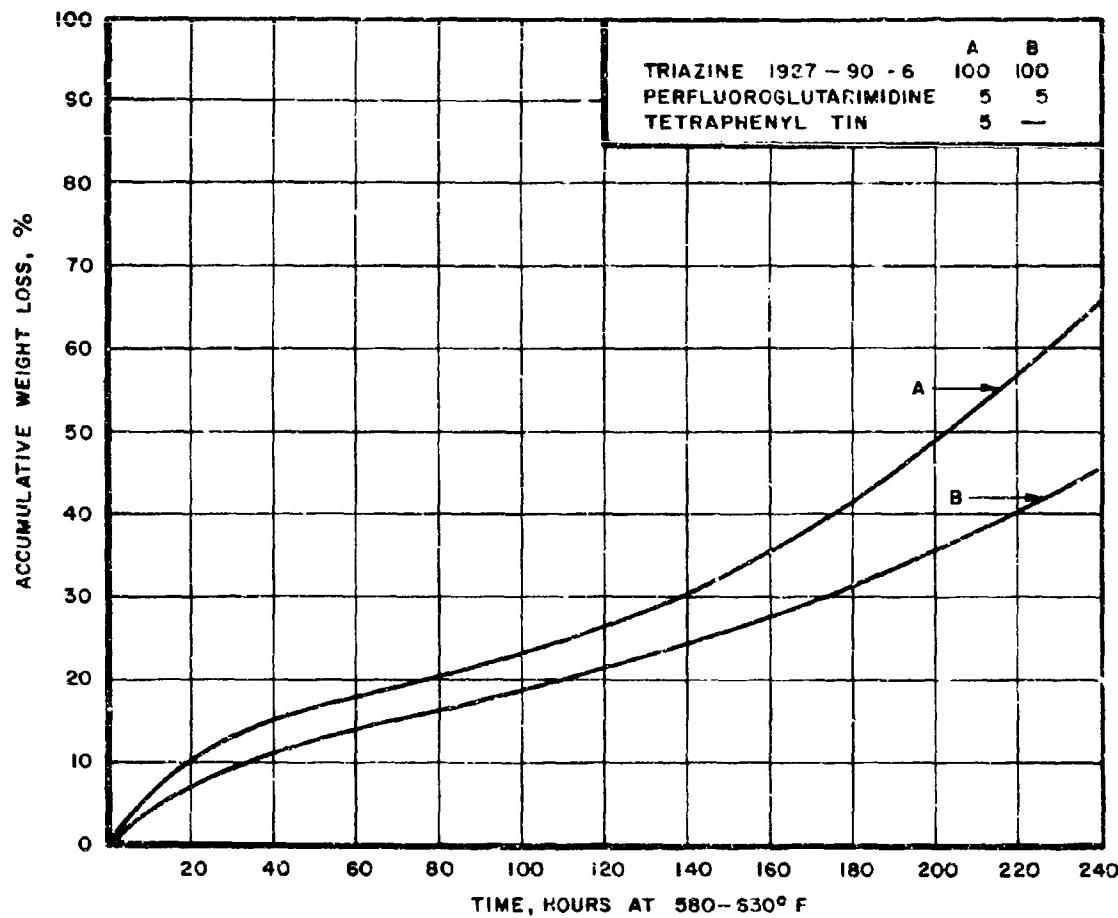


Figure 3. Relative Thermal Stability of Triazine Gum 1927-90-6 Cured with Perfluoroglutarimidine and a Mixture of Perfluoroglutarimidine and Tetraphenyl Tin

TABLE X  
ADDITIONAL TRIAZINE GUM 1927-96-6 COMPOUNDS CURED WITH PERFLUOROGLUTARIMIDINE

(1) Triazine gun preheat treated @ 600° F for 472 hours to remove volatile material.

## SECTION IV

## CURING CHARACTERISTICS OF ADDITIONAL EXPERIMENTAL LOTS OF POLY [PERFLUOROOCTAMETHYLENE (PERFLUOROPROPYL) TRIAZINE] GUMS.

Several compounds were prepared using other samples (Lots 2215-71-1 and 2227-48-1) of the poly[perfluorooctamethylene (perfluoropropyl) triazine] gum supplied later in the program in further exploiting tetraphenyl tin and perfluoroglutarimidine as curing agents. The compositions prepared and the curing cycles used are summarized in Table XI.

The results were unsatisfactory. The tetraphenyl tin compounds based on these later experimental gums yielded undercured products containing voids. Although satisfactory vulcanizates were obtained using perfluoroglutarimidine, these vulcanizates became porous within approximately 16 hours at 600°F.

TABLE XI

## ADDITIONAL EXPERIMENTAL POLY [PERFLUOROOCTAMETHYLENE (PERFLUOROPROPYL) TRIAZINE] GUM-CURING AGENT COMPOUNDS EVALUATED

Triazine 2215-71-1	100	-	-	100	-
Triazine 2227-48-1	-	100	100	-	100
Tetraphenyl Tin	5	5	10	-	-
Perfluoroglutarimidine	-	-	-	10	10
Press-Cure Time @ 320° F, Hours	20	20	20	2	2

## SECTION V

CURING CHARACTERISTICS OF EXPERIMENTAL TRIAZINE GUMS  
CONTAINING n-PERFLUOROPROPYLENE CYANIDE PENDANT GROUPS

A couple of samples of poly[perfluorooctamethylene (perfluoropropyl) triazine] gum containing theoretically 20 mole % n-perfluoropropylene cyanide pendant groups were utilized when further attempting to obtain a thermally stable cured product. One sample of this particular species of the triazine gum (Lot 2227-49 1) (Reference 3) proved to be very reactive towards many of the compounds previously screened. In addition to obtaining cured samples using tetraphenyl tin and perfluoroglutarimidine, adequate states of cure were obtained with tetraphenyl silicon, perfluoro sebacamide and mixtures of the latter two compounds. A structurally sound vulcanizate was also obtained with tributyl antimony oxide, one of the two new candidate curing agents evaluated. Perfluoro sebacic acid, the other new candidate curing agent, appeared to degrade this polymer. Although perfluoro sebacamide proved to be an effective compound for curing the other sample of modified triazine gum (Lot 15B), (Reference 3) satisfactory vulcanizates were not obtained using tetraphenyl tin. The compositions of the compounds prepared with these elastomer samples are given in Table XII.

The cured products proved to be inadequately stable at 600°F. The perfluoroglutarimidine and the perfluoro sebacamide vulcanizates became spotted with pinholes within 16 hours. Vulcanizates obtained using tributyl antimony oxide, tetraphenyl silicon, tetraphenyl tin, perfluoro sebacamide and mixtures of the latter two compounds lost weight rapidly at 600°F (Figure 4).

TABLE XII  
n-PERFLUOROPROPYLENE CYANIDE MODIFIED POLY [PERFLUOROOCOTAMETHYLENE (PERFLUOROPROPYL)<sub>n</sub>-TRIAZINE] GUM-CURING AGENT COMPOUNDS EVALUATED

	Press Cure Time @ 320° F., Hours	1	1	1	1	19	2	4	2 1/2	2	2	20
Triazine 2227-49-1		100	100	100	100	100	100	100	100	-	-	-
Triazine 15B		-	5	-	-	-	-	-	100	100	100	100
Perfluorobenzimidine		10	-	5	-	-	-	-	-	20	-	-
Perfluoroglutarimidine		-	-	-	10	-	-	-	-	-	-	-
Perfluoroisobutyric Acid		-	-	-	-	-	10	-	-	-	-	-
Tetraphenyl Tin		-	1	2.5	-	10	-	-	-	-	10	20
Tetraphenyl Silicon		-	-	-	-	-	10	-	-	-	-	-
Tributyl Antimony Oxide		-	-	-	-	-	-	5	-	-	-	-

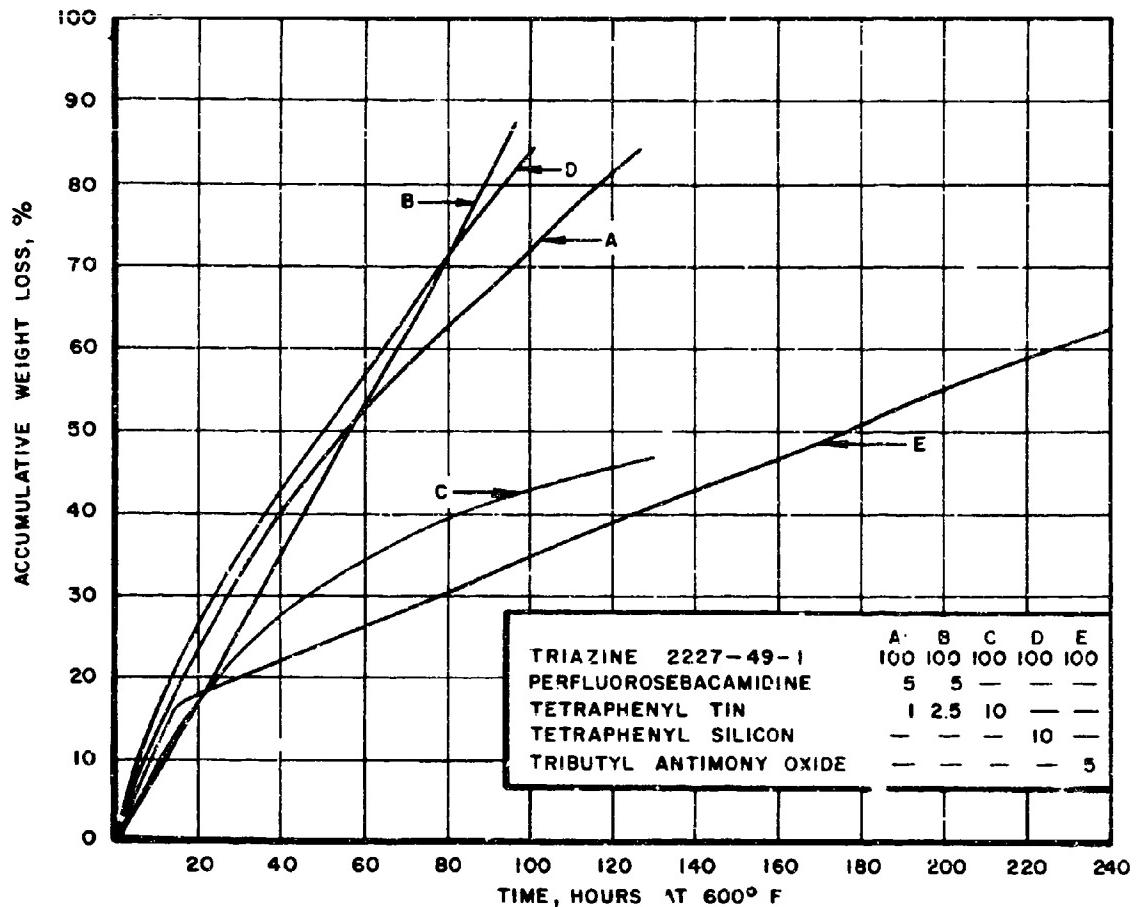


Figure 4. Relative Thermal Stability of Several Chemically Cured Vulcanizates Based on n-Perfluoropropylene Cyanide Modified Triazine Gum 2227-49-1

## SECTION VI

**ASBESTOS REINFORCED CHEMICALLY CROSSLINKED  
TRIAZINE VULCANIZATES**

Several gum vulcanizates and asbestos extended chemically cured compounds based on the n-perfluoropropylene cyanide modified and unmodified species of the poly[perfluorooctamethylene (perfluoropropyl) triazine] gums were prepared to determine the overall merits of asbestos as a reinforcing agent and to obtain 500°F aging data.

The compositions of the vulcanizates based on the poly [perfluoroctamethylene (perfluoropropyl) triazine ] gums and the tensile data obtained are presented in Table XIII. Comparable data and the compositions of vulcanizates based on the n-perfluoropropylene cyanide modified poly[ perfluoroctamethylene (perfluoropropyl) triazine] gums are shown in Table XIV.

As is apparent from the data, original tensile strengths of the asbestos reinforced vulcanizates were 200 to 300 psi greater than the unreinforced gum vulcanizates. The tensile strength of all the vulcanizates increased following various periods of exposure at 500°F. These gains in tensile strength were most likely due to thermally induced polymer rearrangements and were accompanied by losses in weight.

Contrary to previous results, the asbestos filler for the most part had an adverse effect on thermal stability (Figures 5 and 6). The rates of weight loss were generally greater for the asbestos reinforced vulcanizates than for the unreinforced cured gums as well as the uncured gums. As the plotted weight loss data show, vulcanizates based on the asbestos reinforced poly[perfluoroctamethylene (perfluoropropyl) triazine] gum were more thermally resistant than the asbestos reinforced vulcanizates based on the n-perfluoropropylene cyanide modified poly[perfluoroctamethylene (perfluoropropyl) triazine] gum. Although the rates of weight loss decreased with time, the former vulcanizates lost from 21 to 26 percent of their weight within a period of 500 hours at 500°F.

TABLE XIII

**TENSILE PROPERTIES OF ASBESTINE 3X EXTENDED POLY  
[ PERFLUOROCOTAMETHYLENE (PERFLUOROPROPYL)  
TRIAZINE ] VULCANIZATES**

Triazine 2215-71-1	100	100	-	-	-
Triazine 2227-48-1	-	-	100	100	-
Triazine 1927-90-6	-	-	-	-	100
Perfluoroglutarimidine	10	10	10	10	-
Tetraphenyl Tin	-	-	-	-	10
Asbestine 3X	-	20	-	20	20
<b>Original</b>					
Tensile Strength, psi	240	445	230	425	730
Elongation, %	210	175	105	105	600
<b>After 16 hours @ 500° F</b>					
Tensile Strength, psi	365	730	360	750	1005
Elongation, %	165	130	200	145	225
<b>After 72 hours @ 500° F</b>					
Tensile Strength, psi	440	770	335	820	540
Elongation, %	195	100	185	140	295
<b>After 10 days @ 500° F</b>					
Tensile Strength, psi	490	885	355	925	-
Elongation, %	115	115	200	115	-
<b>After 20 days @ 500° F</b>					
Tensile Strength, psi	-	-	-	-	505
Elongation, %	-	-	-	-	230
<b>After 24 days @ 500° F</b>					
Tensile Strength, psi	285	940	225	845	-
Elongation, %	175	130	165	130	-

TABLE XIV

TENSILE PROPERTIES OF ASBESTINE 3X EXTENDED VULCANIZATES  
BASED ON POLY[PERFLUOROOCTAMETHYLENE (PERFLUOROPROPYL)  
TRIAZINE] GUMS CONTAINING n-PERFLUOROPROPYLENE CYANIDE  
PENDANT GROUPS

Triazine 15B	100	100	..	-
Triazine 2227-49-1	-	-	100	100
Perfluorosebacamidine	10	10	10	10
Asbestine 3X	-	20	-	20
<b>Original</b>				
Tensile Strength, psi	375	675	75	1035
Elongation, %	135	125	1	105
<b>After 16 hours @ 500° F</b>				
Tensile Strength, psi	510	630	710	1375
Elongation, %	30	80	130	115
<b>After 72 hours @ 500° F</b>				
Tensile Strength, psi	445	865	335	1275
Elongation, %	125	80	70	75
<b>After 10 days @ 500° F</b>				
Tensile Strength, psi	335	520	- (1)	1400
Elongation, %	135	95	-	65

(1) This vulcanizate after 10 days at 500° F contained numerous bubbles.

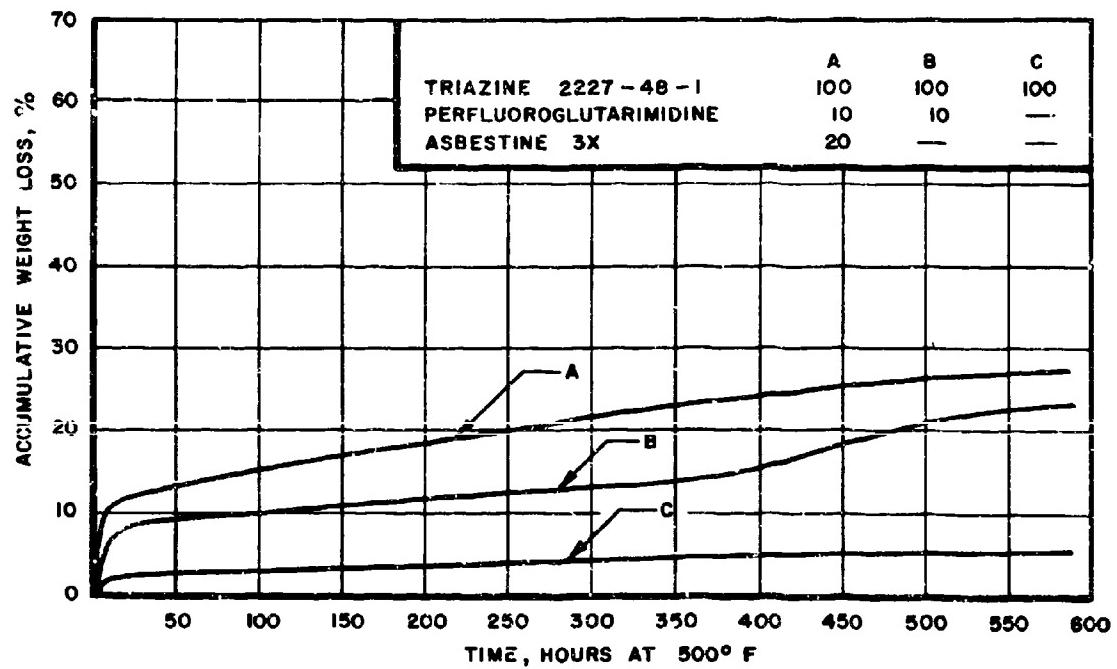
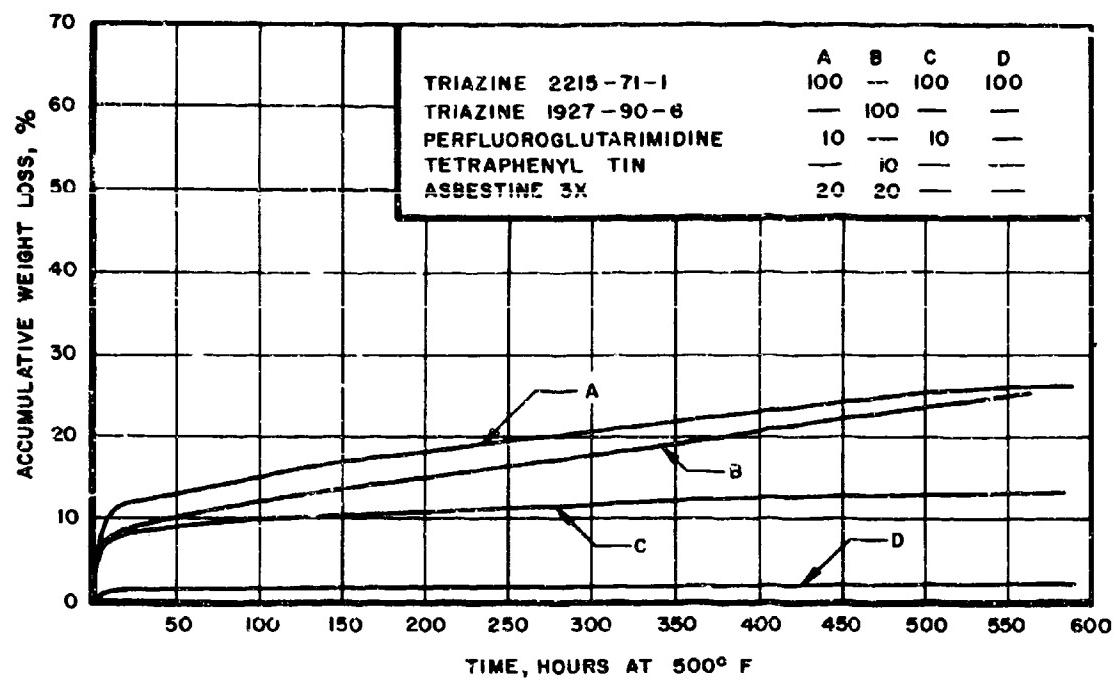


Figure 5. Effect of Asbestine 3X on the Thermal Stability of Vulcanizates Based on Poly[perfluorooctamethylene (perfluoropropyl) Triazine] Gums

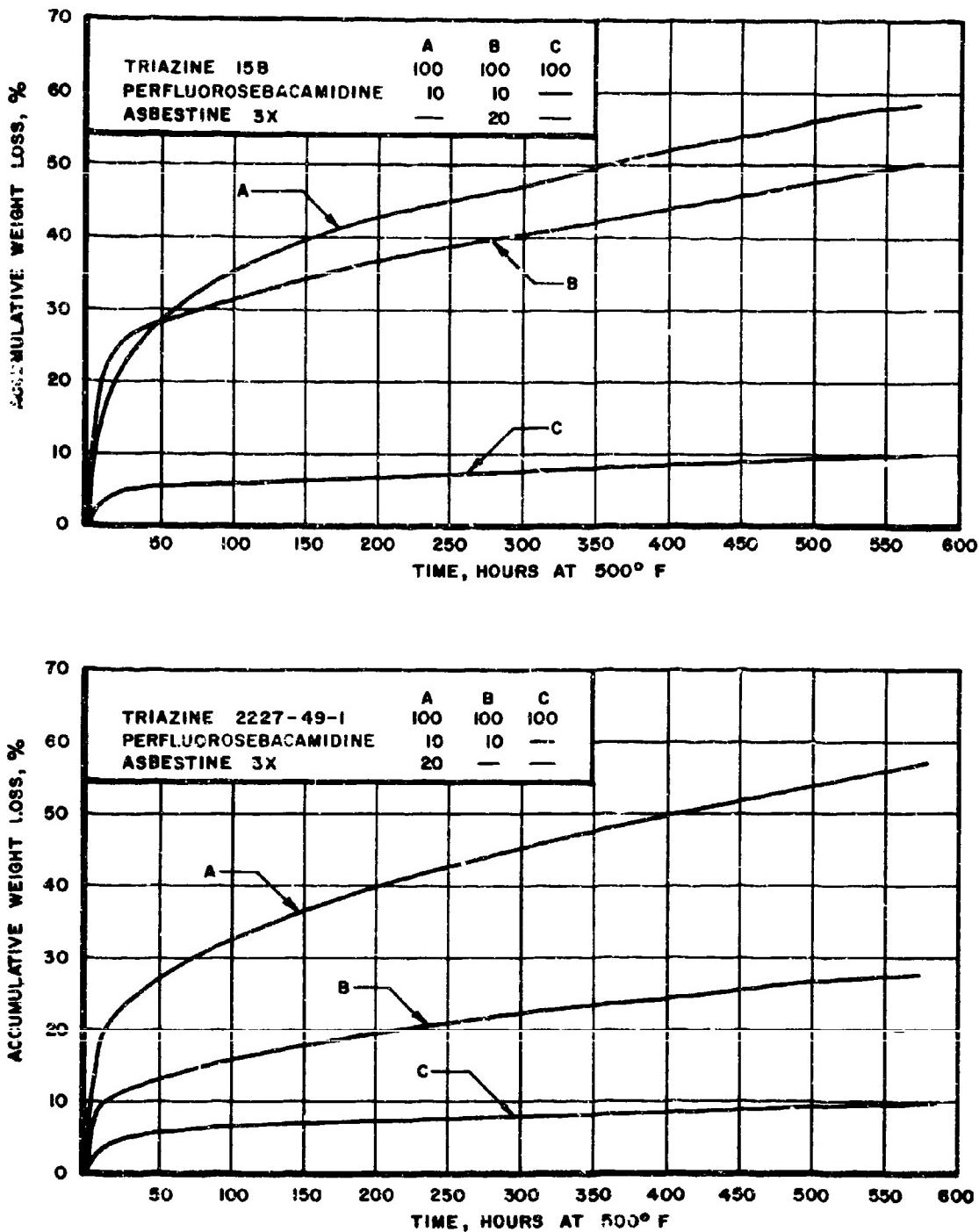


Figure 6. Effect of Asbestine 3X on the Thermal Stability of Vulcanizates Based on Poly [perfluoroctamethylene (perfluoropropyl) Triazine] Gums Containing u-Perfluoropropylene Cyanide Pendant Groups

## SECTION VII

### SUMMARY AND CONCLUSIONS

This internal research program was an endeavor to obtain suitable modifiers for cross-linking and reinforcing the triazine elastomers. Several samples of three different species of the triazine polymer were involved in this exploratory compounding research program.

The selected candidate reinforcing materials were screened using samples of poly[perfluoropropylene (perfluoropropyl) triazine] gum. All of the materials evaluated (carbon blacks, several microfibrous products, metal oxide powders, mineral fillers and a perfluorinated resinous aromatic polymer bolstered the strength of the samples of poly[perfluoropropylene (perfluoropropyl) triazine] gum used in the screening experiments. The most favorable results were obtained with the asbestos fibrous fillers. Increases in strength were obtained using as much as 20 phr of a micro-fibrous asbestos filler (Asbestine 3X) at no loss in thermal stability and nominal reductions in elongation. In contrast to these results, the gains in strength realized with 15 phr of the other candidate reinforcing agents (in the case of Cab-O-Sil only 10 phr) were accompanied by significant reductions in elongation and/or increased rates of thermal decomposition.

An approach involving the use of a poly [perfluoropropylene (perfluoropropyl) triazine] resin in conjunction with asbestos filler lead to thermally cured elastomeric poly[perfluoropropylene (perfluoropropyl) triazine] products possessing good strength (565 to 900 psi). Unfortunately the vulcanizates obtained using this combination of additives proved to be extremely unstable to heat.

Experimental lots of poly[perfluoroctamethylene (perfluoropropyl) triazine] gum and n-perfluoropropylene cyanide modified poly[polyfluoroctamethylene (perfluoropropyl) triazine] gum were used for investigating the compounds selected for evaluation as curing agents. There were considerable differences in the reactivities of different lots of each of these species of the triazine polymer. While tetraphenyl tin, tetraphenyl lead and perfluoroglutarimidine proved to effective curing agents for one lot of the poly[perfluoroctamethylene (perfluoropropyl) triazine] gum, other lots of this gum could only be cured to an adequate degree using perfluoroglutarimidine. Similarly, with one lot of the poly[perfluoroctamethylene (perfluoropropyl) triazine] gum containing theoretically 20 mole percent n-perfluoropropylene cyanide pendant groups good states of cure were obtained using tetraphenyl silicon, tetraphenyl tin, tributylantimony oxide, perfluoroglutarimidine and perfluorosebacamidine whereas another sample of the modified gum could only be satisfactorily cured using the latter compound. All of these vulcanizates had low tensile strengths (245 to 325 psi) and proved to be inadequately stable in the 580° to 630°F temperature region.

Additional vulcanizates based on several of the above systems were prepared to obtain 500°F aging data as well as to determine further the merits of Asbestine 3X as a reinforcing agent. The asbestos filler improved the tensile strength of the vulcanizates but contrary to the results of initial screening experiments most generally hastened thermal decomposition. The results of 500°F aging showed the asbestos reinforced perfluoroglutarimidine and tetraphenyl tin vulcanizates based on the unmodified triazine gums to be more thermally stable than the perfluorosebacamidine cured asbestos extended n-perfluoropropylene cyanide modified poly[perfluoroctamethylene (perfluoropropyl) triazine] gums. The perfluoroglutarimidine and tetraphenyl tin asbestos reinforced poly[perfluoroctamethylene (perfluoropropyl) triazine] gums lost between 8.5 to 11.5 percent weight during the first 24 hours at 500°F. Although the rates of weight lost rapidly diminished on prolonged exposure these vulcanizates lost from 21 to 26 percent of their weight within 500 hours at 500°F and were continuing to lose weight beyond the 500-hour period at rates ranging from 0.02 to 0.03%/hour.

The mechanism whereby cured triazine products have been obtained using certain organometallic compounds and ammoniated perfluorinated dinitriles is not thoroughly understood. Although the results of experiments by Hooker Chemical with model compounds have shown certain organometallic compounds to be effective catalysts for trimerizing nitrile bearing compounds, the crosslinking reaction involving polymer appears to be more complex than the conversion of nitrile to triazine crosslinks. This conclusion is supported by the fact that an all perfluoropropyl pendant as well as perfluoropropylene cyanide modified poly[perfluoroctamethylene (perfluoropropyl) triazine] gum have been vulcanized using organometallic compounds. The triazine elastomer is a very difficult polymer to synthesize, and the possibility that some of the current products inadvertently contain in-chain reactive sites which result from incomplete reaction during the ring closure step may account for the decided differences in the reactivities of different lots of the same species of the triazine polymer towards the organometallic type curing agents. On the other hand, there is strong evidence that cured products are obtained (using the non-metallic compounds) through disruption of the basic polymer structure. In working with model compounds, Dr. H. C. Brown (Reference 3) found that nucleophiles such as  $\text{NH}_3$ , which is liberated by the ammoniated perfluorodinitrile curing agents during the curing process, do indeed attack the triazine structure splitting out volatile chain fragments. The results of this internal research more or less corroborate Brown's work and indicate that the ammonia liberated from the non-metallic curing agents is retained and causes continual rearrangement of the cured polymer network, and in the process, polymer degradation.

The results of this work in general suggest that polymer with other functional groups may offer a better chance of success in attaining crosslinked products with the required thermal resistance. However, because of the questionable structural features of the lots of products involved in the current work there is some uncertainty regarding the adequacy of the current types of triazine polymer. In view of the foregoing uncertainty and since it is possible to prepare linear polymer by limiting the molecular weight, further exploratory work with the current species of the triazine polymer appears to be warranted, and is required to establish the future course to be pursued in the synthesis area.

SECTION VIII  
REFERENCES

1. Dorfman, E. and Emerson, W. E., "Perfluoroalkylene Triazine Elastomeric Polymers," Air Force Materials Laboratory, Technical Documentary Report No. ML-TDR-64-249, Part I, August 1964.
2. Griffin, W. R., "Perfluoroalkyl Triazine Elastomers," Aeronautical Systems Division, Technical Documentary Report No. ASD-TDR-62-1114, June 1963.
3. Dorfman, E. and Emerson, W. E., "Perfluoroalkylene Triazine Elastomeric Polymers," Air Force Materials Laboratory, Technical Documentary Report No. ML-TDR-64-249, Part II, August 1966. (See Subcontract Report Section by H. C. Brown)

## UNCLASSIFIED

Security Classification

## DOCUMENT CONTROL DATA - R&amp;D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1. ORIGINATING ACTIVITY (Corporate author) Air Force Materials Laboratory Research and Technology Division Air Force System Command Wright-Patterson Air Force Base, Ohio	2a. REPORT SECURITY CLASSIFICATION Unclassified
	2b. GROUP

## 3. REPORT TITLE

## CROSSLINKING AND FILLER REINFORCEMENT OF PERFLUOROALKLENE TRIAZINE ELASTOMERS

## 4. DESCRIPTIVE NOTES (Type of report and inclusive dates)

Work accomplished during the period April 1964 to September 1966

## 5. AUTHOR(S) (Last name, first name, initial)

Graham, T. L.

6. REPORT DATE May 1967	7a. TOTAL NO. OF PAGES 36	7b. NO. OF REFS 3
8a. CONTRACT OR GRANT NO.	9a. ORIGINATOR'S REPORT NUMBER(S) AFML-TR-66-402	
b. PROJECT NO. 7340		
c. Task No. 734005	9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report) d.	

## 10. AVAILABILITY/LIMITATION NOTICES

This document may be further distributed by any holder only with specific prior approval of the Elastomers and Coatings Branch, (MANE), Nonmetallic Materials Division, Air Force Materials Laboratory, Wright-Patterson Air Force Base, Ohio 45433.

11. SUPPLEMENTARY NOTES	12. SPONSORING MILITARY ACTIVITY Air Force Materials Laboratory Research and Technology Division Air Force System Command Wright-Patterson Air Force Base, Ohio
-------------------------	---

## 13. ABSTRACT

This exploratory compounding development program was conducted for the purpose of obtaining suitable modifiers for the crosslinking and reinforcing of triazine elastomers. The reinforcing effects of a variety of finely divided materials are compared. Data are presented showing the effect of concentration of the asbestos type filler (which yielded the most promising results) on the physical properties and thermal stabilities of a poly [perfluoropropylene (perfluoropropyl) triazine] gum and a blend comprised of equal parts of the gum and a poly [perfluoropropylene (perfluoropropyl) triazine] resin. Several organometallic and organic compounds were found to be effective crosslinking agents for poly [perfluorooctamethylene (perfluoropropyl) triazine] and n-perfluoropropylene cyanide modified poly [perfluorooctamethylene (perfluoropropyl) triazine] elastomers. The physical properties and thermal resistance of chemically vulcanized gums and asbestos reinforced vulcanizates based on the n-perfluoropropylene cyanide modified and unmodified poly [perfluorooctamethylene (perfluoropropyl) triazine] gums are discussed in detail.

This abstract may be further distributed by any holder only with specific prior approval of the Elastomers and Coatings Branch, (MANE), Nonmetallic Materials Division, Air Force Materials Laboratory, Wright-Patterson Air Force Base, 45433.

DD FORM 1 JUN 64 1473

UNCLASSIFIED

Security Classification

## UNCLASSIFIED

Security Classification

14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Triazine Elastomers Elastomers Triazine Polymer Crosslinking Agents Compounding Triazine Elastomers						
INSTRUCTIONS						
<p>1. ORIGINATING ACTIVITY: Enter the name and address of the contractor, subcontractor, grantee, Department of Defense activity or other organization (corporate author) issuing the report.</p> <p>2a. REPORT SECURITY CLASSIFICATION: Enter the overall security classification of the report. Indicate whether "Restricted Data" is included. Marking is to be in accordance with appropriate security regulations.</p> <p>2b. GROUP: Automatic downgrading is specified in DoD Directive 5200.10 and Armed Forces Industrial Manual. Enter the group number. Also, when applicable, show that optional markings have been used for Group 3 and Group 4 as authorized.</p> <p>3. REPORT TITLE: Enter the complete report title in all capital letters. Titles in all cases should be unclassified. If a meaningful title cannot be selected without classification, show title classification in all capitals in parentheses immediately following the title.</p> <p>4. DESCRIPTIVE NOTES: If appropriate, enter the type of report, e.g., interim, progress, summary, annual, or final. Give the inclusive dates when a specific reporting period is covered.</p> <p>5. AUTHOR(S): Enter the name(s) of author(s) as shown on or in the report. Enter last name, first name, middle initial. If military, show rank and branch of service. The name of the principal author is an absolute minimum requirement.</p> <p>6. REPORT DATE: Enter the date of the report as day, month, year, or month, year. If more than one date appears on the report, use date of publication.</p> <p>7a. TOTAL NUMBER OF PAGES: The total page count should follow normal pagination procedures, i.e., enter the number of pages containing information.</p> <p>7b. NUMBER OF REFERENCES: Enter the total number of references cited in the report.</p> <p>8a. CONTRACT OR GRANT NUMBER: If appropriate, enter the applicable number of the contract or grant under which the report was written.</p> <p>8b, 8c, &amp; 8d. PROJECT NUMBER: Enter the appropriate military department identification, such as project number, subproject number, system numbers, task number, etc.</p> <p>9a. ORIGINATOR'S REPORT NUMBER(S): Enter the official report number by which the document will be identified and controlled by the originating activity. This number must be unique to this report.</p> <p>9b. OTHER REPORT NUMBER(S): If the report has been assigned any other report numbers (either by the originator or by the sponsor), also enter this number(s).</p> <p>10. AVAILABILITY/LIMITATION NOTICES: Enter any limitations on further dissemination of the report, other than those imposed by security classification, using standard statements such as:</p> <ul style="list-style-type: none"> <li>(1) "Qualified requesters may obtain copies of this report from DDC."</li> <li>(2) "Foreign announcement and dissemination of this report by DDC is not authorized."</li> <li>(3) "U. S. Government agencies may obtain copies of this report directly from DDC. Other qualified DDC users shall request through ."</li> <li>(4) "U. S. military agencies may obtain copies of this report directly from DDC. Other qualified users shall request through ."</li> <li>(5) "All distribution of this report is controlled. Qualified DDC users shall request through ."</li> </ul> <p>If the report has been furnished to the Office of Technical Services, Department of Commerce, for sale to the public, indicate this fact and enter the price, if known.</p> <p>11. SUPPLEMENTARY NOTES: Use for additional explanatory notes.</p> <p>12. SPONSORING MILITARY ACTIVITY: Enter the name of the departmental project office or laboratory sponsoring (paying for) the research and development. Include address.</p> <p>13. ABSTRACT: Enter an abstract giving a brief and factual summary of the document. Indicate of the report, even though it may also appear elsewhere in the body of the technical report. If additional space is required, a continuation sheet shall be attached.</p> <p>It is highly desirable that the abstract of classified reports be unclassified. Each paragraph of the abstract shall end with an indication of the military security classification of the information in the paragraph, represented as (TS), (S), (C), or (U).</p> <p>There is no limitation on the length of the abstract. However, the suggested length is from 150 to 225 words.</p> <p>14. KEY WORDS: Key words are technically meaningful terms or short phrases that characterize a report and may be used as index entries for cataloging the report. Key words must be selected so that no security classification is required. Identifiers, such as equipment model designation, trade name, military project code name, geographic location, may be used as key words but will be followed by an indication of technical context. The assignment of links, rules, and weights is optional.</p>						

UNCLASSIFIED

Security Classification